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IS 4525 (1994): 4-Aminoacetanilide, Technical [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]

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भारतीय मानक

4-एमिनोएसिटेनीलाइड, तकनीकी – विशिष्टि

(पहला पुनरीक्षण)

Indian Standard

4-AMINOACETANILIDE, TECHNICAL — SPECIFICATION

(First Revision)

UDC 667·281·79 : 547·556·33

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

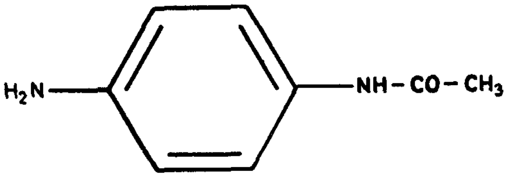
April 1994

Price Group 3

FOREWORD

This Indian Standard (First Revision) was adopted by Bureau of Indian Standards after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

4-Aminoacetanilide ($C_8H_{10}N_2O$) is an important dye intermediate used in the manufacture of azo dyes. It is also used in the manufacture of pharmaceuticals. This standard covers only in the base, though it is also marketed as the hydrochloride. It has the following structural formula:



4-AMINOACETANILIDE

Molecular Mass = 150·18

CAS registry number [122-80-5]

This standard was first published in 1968. The committee responsible for its preparation decided to update the standard in light of the experience gained. In this version, the requirement of impurity such as 4-Nitroacetanilide has been stipulated. The requirements for melting point and solubility in hot water have been deleted. The requirement for solubility in dilute hydrochloric acid has been modified. The 'Title' has also been changed as 4-Aminoacetanilide.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

4-AMINOACETANILIDE, TECHNICAL — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements and methods of sampling and test for 4-aminoacetanilide.

2 NORMATIVE REFERENCES

The following Indian Standards contain provisions which through reference in the text, constitute provisions of this standard. At the time of publication the editions indicated were valid. All standards are subject to revision and parties to agreement, based on the standard are encouraged to investigate the possibility of applying the most recent editions of the standards Indicated below:

IS No.	*Title*
1070 : 1992	Water for general laboratory use (*third revision*)
1260 (Part 1) : 1973	Pictorial marking for handling and labelling of goods: Part I Dangerous goods (*first revision*)
2552 : 1989	Steel drums (galvanized and ungalvanized) (*third revision*)
5299 : 1969	Methods for sampling and test for dye intermediates

3 REQUIREMENTS

3.1 Description

The material shall be in the form of colourless to slightly pinkish crystalline powder.

3.2 The material shall also comply with the requirements given in Table 1.

4 PACKING AND MARKING

4.1 Packing

Unless otherwise agreed, the material shall be suitably packed either in wooden barrels, multiwalled paper sacks or suitable drums.

4.2 Marking

The containers shall be securely closed and shall be marked with the following information:

a) Name of the material,
b) Indication of the source of manufacture,
c) Net mass of material,
d) Batch or lot number, and
e) Month and year of manufacture.

4.2.1 The containers shall also be labelled with Fig. 5 of IS 1260 (Part 1) : 1973 containing the following minimum cautionary notice:

"WARNING : AVOID CONTACT WITH SKIN, DO NOT HEAT, EMITS TOXIC FUMES"

4.3 BIS Certification Marking

The containers may also be marked with the Standard Mark.

4.3.1 The use of the Standard Mark is governed by the provisions of Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5 SAMPLING

5.1 The method of drawing representative samples of the material shall be as prescribed in Annex C.

6 TEST METHODS

6.1 Tests shall be carried out according to the methods prescribed in col 4 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, 'pure chemicals' and distilled water (*see* IS 1070 : 1992), shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

Table 1 Requirements for 4-Aminoacetanilide, Technical

(*Clauses* 3.2, 6.1 *and* C-6.2)

Sl No. (1)	Characteristics (2)	Requirement (3)	Method of Test, Ref to (4)
i)	Assay, percent by mass, *Min*	92	Annex A
ii)	Matter insoluble in dilute hydrochloric acid, percent by mass, *Max*	0·3	10 of IS 5299 : 1969
iii)	4-Nitroacetanilide, percent by mass, *Max*	0·2	Annex B

ANNEX A

[*Table* 1, *Item* (i)]

DETERMINATION OF ASSAY

A-0 OUTLINE OF THE METHOD

A known amount of the material is dissolved in hydrochloric acid and diazotised with standard sodium nitrite solution, using starch-iodide test paper as indicator.

A-1 REAGENTS

A-1.1 Hydrochloric Acid — 30 percent (*w*/*v*).

A-1.2 Standard Sodium Nitrite Solution — 1 N, freshly standardized.

A-1.3 Starch-Iodide Test Paper

A-2 PROCEDURE

Weigh accurately about 5 g of the sample and transfer to a one-litre beaker. Add 400 ml of water and dissolve at room temperature. Then add while stirring, 25 ml of hydrochloric acid (a solution of *p*-aminoacetanilide hydrochloric is readily hydrolysed to *p*-phenylenediamine if warmed, with consequent high results). Add ice cold water to cool to 20°C. While stirring mechanically, titrate with standard sodium nitrite solution which is added through a thistle funnel the end of which is well inside the solution. Add standard sodium nitrite solution as rapidly as it is consumed. The end point is reached when an immediate blue coloured ring appears on starch-iodide test paper which can be obtained repeatedly during a period of 10 minutes without further addition of sodium nitrite. Note the volume of standard sodium nitrite solution required for titration.

A-3 CALCULATION

Assay, percent by mass (calculated in molecular mass 150·18) $= \dfrac{V \times 15{\cdot}01 \times N}{M}$

where

V = volume in ml of standard sodium nitrite solution required for titration,

N = normality of standard sodium nitrite solution, and

M = mass in g of the sample taken for the test.

ANNEX B

[*Table* 1, *Item* (iii)]

DETERMINATION OF 4-NITROACETANILIDE

Solution : 2 percent in Acetone + Ammonia (8 + 2)

Application : 10 µl = 200 µg

Test substance : 4-Nitroacetanilide

Soln : 0·04 percent in acetone

App : 5µl = 0·2 µg = 0·1 percent

10 µl = 0·4 µg = 0·2 percent

TLC : Silica gel G

Developer : Benzene : Ethanol (80 : 20). Ammonia atmosphere

Temperature : Room temperature

Run : 15 cm

Detection : Spray with spraying reagent which is prepared as follows:

1) 0·5 percent *p*-Dimethylaminobenzaldehyde in equal proportion of 5N Hydrochloric acid and methanol.
2) 10 percent stannous chloride in 5N hydrochloric acid
3) Mix (1) and (2) in equal proportion which is ready as spraying reagent.

ANNEX C

(*Clause* 5.1)

SAMPLING OF 4-AMINOACETANILIDE, TECHNICAL

C-1 GEENRAL REQUIREMENTS OF THE SAMPLING

C-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

C-1.1 Samples shall be taken at a place protected from damp air, dust and soot.

C-1.2 Sampling instrument shall be clean and dry.

C-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument, and the containers for a sample from adventitious contamination.

C-1.4 To draw a representative sample, the contents of each container, selected for sampling, shall be mixed, as thoroughly as possible, by suitable means.

C-1.5 The samples shall be placed in clean, dry and air-tight glass or other suitable containers on which the material has no action.

C-1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.

C-1.7 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling, year of manufacture, and other important particulars of the consignment.

C-1.8 Samples shall be stored in a cool and dry place.

C-2 SAMPLING INSTRUMENT

C-2.1 The sampling instrument is a closed type, sampling tube, undivided (Fig. 1), consisting of two concentric cylindrical tubes made of a metal which is not affected by the material being sampled (preferably of stainless steel) closely fitting into each other throughout their entire length so that one tube can be rotated within the other, a suitable handle being provided for the purpose. Longitudinal openings of about one-third the circumference are cut in both tubes throughout their length. In one position the two openings coincide and admit the material into the hollow inner tube. By rotating the inner tube through 180° the opening is tightly closed and a 'core' of material being enclosed therein may be withdrawn. This type of sampler is usually provided with locking arrangement so that the tubes are held together in any desired position. The outer tube is provided with a sharp conical end to facilitate penetration but the base of the cone shall be closed so that no material is entrapped in this portion. The height of the cone shall be equal to its base diameter. The whole instrument shall be of sufficient length to penetrate an entire diagonal of the container being sampled. The diameter of the inner cylindrical space may vary from 20 to 40 mm proportionately to the length. A length of 1 500 mm and a diameter of 30 mm is satisfactory for most needs.

C-2.1.1 *Use of Sampling Instrument*

The instrument is inserted in closed position in an oblique direction till it touches bottom. The material is admitted by rotating and opening the tubes and finally closing them, withdrawing the sample in this process. In case the minimum quantity of material required for test from each container is more than the capacity of the instrument, further 'cores' shall be taken from different parts of the same container such that they are at least 75 mm from the wall of the container in case of kegs, drums, bags, etc, and 25 mm in case of small containers. In all cases the instrument shall be inserted till it touches bottom so that an entire cross-section is withdrawn.

C-3 SCALE OF SAMPLING

C-3.1 Lot

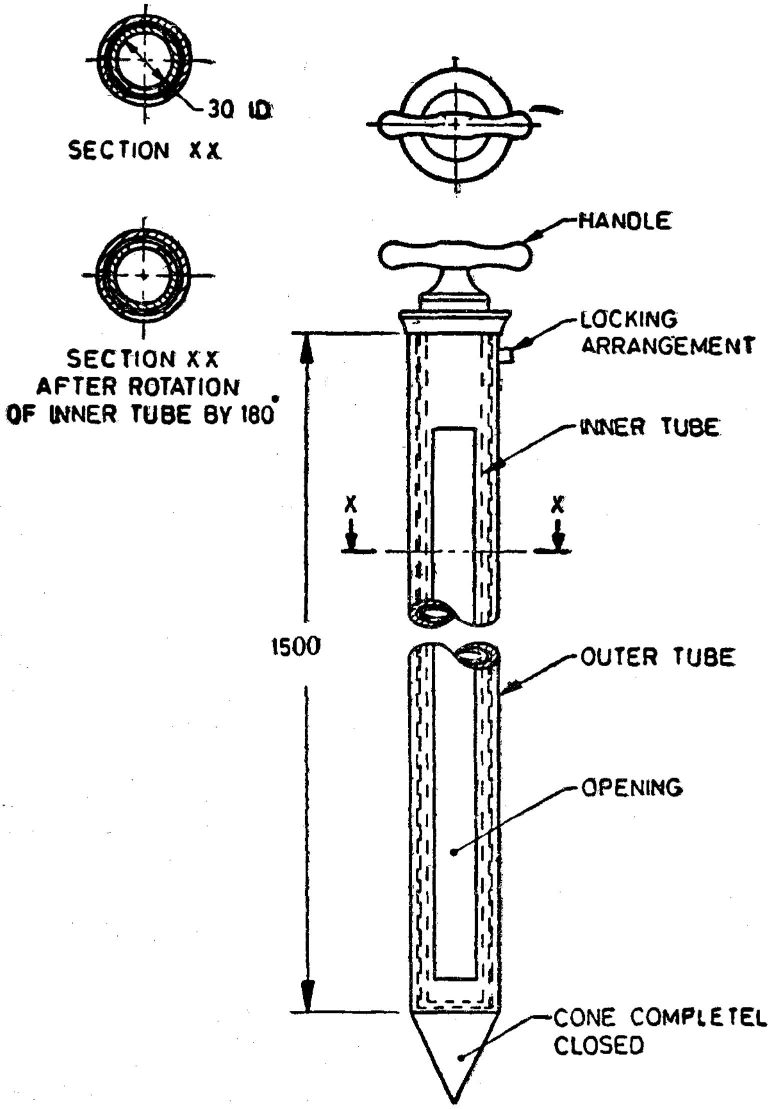
All the containers in a single consignment of the material from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of containers pertaining to different batches of manufacture, then the containers belonging to the same batch of manufacture shall be grouped together and each such group shall constitute a separate lot.

C-3.2 For ascertaining the conformity of the lot to the requirements of this specification, tests shall be carried out for each lot separately. The number (n) of containers to be selected for drawing the sample shall depend upon the size (N) of the lot and shall be in accordance with Table 2.

Table 2 Number of Containers to be Selected for Sampling

Lot Size	No of Containers to be Selected
(1	(2)
4 to 15	3
16 „ 40	4
41 „ 65	5
66 „ 110	6
111 „ 200	7
201 and above	8

NOTE — When the size of the lot is three or less, all the containers shall be sampled.



All dimensions in millimetres.

FIG. 1 CLOSED TYPE SAMPLING TUBE, UNDIVIDED

C-3.3 These containers shall be selected at random from the lot and to ensure the randomness of selection, random number tables shall be used. In case, such tables are not available, the following procedure may be adopted:

Starting from any container, count them in one order as 1, 2, 3 ..... up to *r* and so on, where *r* is the integral part of *N*/*n* (*N* being the lot size and *n* being the number of containers to be selected). Every *r*th container thus counted shall be withdrawn to give sample for test.

C-4 TEST SAMPLE AND REFEREE SAMPLE

C-4.1 From each of the containers selected as in C-3.2, draw with an appropriate sampling implement small portions of the material from different parts of the container. The total quantity so drawn from each of the containers shall be a little more than thrice the quantity required for testing purposes as indicated in C-5.

C-4.2 Mix thoroughly all the portions of the material drawn from the same container to give a representative sample for the container.

C-4.3 From the samples (*see* **C-4.2**) representing different containers selected in **C-3.2**, a small but equal quantity of material shall be taken and thoroughly mixed to form a composite sample, which shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee.

C-4.4 The remaining portion of the material in each sample (*see* C-4.2) from each different container shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the *n* containers selected shall be for the purchaser, another for the supplier and the third for the referee.

C-4.5 All the individual and composite samples shall be transferred to separate container. These containers shall be sealed air-tight with stoppers and labelled with full identification particulars given in **C-1.7**. Care shall be taken not to expose the samples to acid and alkali fumes.

C-4.6 The referee samples consisting of a composite sample and a set of *n* individual samples, shall bear the seals of both the purchaser and the supplier and shall be kept at a place agreed to between the two. This shall be used in case of any dispute between the two.

C-5 NUMBER OF TESTS

C-5.1 Tests for the determination of assay and melting point shall be conducted on each of the individual samples (*see* **C-4.4**) separately.

C-5.2 Tests for the determination of matter insoluble in dilute hydrochloric acid and in hot water shall be conducted on the composite sample (*see* **C-4.3**).

C-6 CRITERIA FOR CONFORMITY

C-6.1 For Individual Samples

The lot shall be declared as conforming to the requirements of assay and melting point, if each of the individual test results as obtained in **C-5.1** satisfy the corresponding requirements given in Table 1.

C-6.2 For Composite Sample

The lot shall be declared as conforming to the requirements of matter insoluble in dilute hydrochloric acid and in hot water, if the test results as obtained in **C-5.2** satisfy the corresponding requirement given in Table 1.

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